FA4869-08-1-4040

Title:
Nano shape memory alloy composite development and applications

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Date:
Jan.27, 2010

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Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
28 JAN 2010	**FInal**	**31-01-2008 to 31-01-2009**

4. TITLE AND SUBTITLE
Nano shape memory alloy composite development and applications

5a. CONTRACT NUMBER
FA48690814040

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Hiroyuki Kato

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Hokkaido University,N13W8 Kita-ku,Sapporo 060-8628,Japan,JP,060-8628

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AOARD, UNIT 45002, APO, AP, 96337-5002

10. SPONSOR/MONITOR'S ACRONYM(S)
AOARD

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
AOARD-084040

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The subject of this study is to process nano metallic fibers by physical method; the fibers will be prepared by ion-sputtering of target material on porous alumina membrane. As compared to conventional chemical method, e.g. the reductive method of nickel nano powders, the physical method has advantages such that the diameter of metallic fibers is controllable by changing the size of holes of the template, and it can be applied to any kind of materials as long as sputtering deposition is possible. The present target material is nickel-titanium shape memory alloy (SMA) of nearly equiatomic composition (50atmic percent of titanium). In this study I have succeeded to get nano-sized columnar crystal grown on the template. I have also found that the columnar crystal is easy to be disintegrated into nano particles and fibers by ultrasonic wave. The crystal structure and thermal property of the present nano SMA fibers were examined; the results show they are almost identical to the bulk mother alloys.

15. SUBJECT TERMS
Smart Structures, Shape Memory Alloys

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
unclassified	**unclassified**	**unclassified**	**Same as Report (SAR)**	**5**	

Abstract

The subject of this study is to process nano metallic fibers by physical method; the fibers will be prepared by ion-sputtering of target material on porous alumina membrane. As compared to conventional chemical method, e.g. the reductive method of nickel nano powders, the physical method has advantages such that the diameter of metallic fibers is controllable by changing the size of holes of the template, and it can be applied to any kind of materials as long as sputtering deposition is possible.

The present target material is nickel-titanium shape memory alloy (SMA) of nearly equiatomic composition (50atmic percent of titanium). In this study I have succeeded to get nano-sized columnar crystal grown on the template. I have also found that the columnar crystal is easy to be disintegrated into nano particles and fibers by ultrasonic wave. The crystal structure and thermal property of the present nano SMA fibers were examined; the results show they are almost identical to the bulk mother alloys.

- ## Results

This study is a part of the collaborative work “Development of nano SMA/SMP fiber composite material and applications”, with U of Washington and U of British Columbia teams. In this work I am responsible for supplying nano SMA fibers to the other members in collaboration, who will utilize the fibers for further processing.

As the result of the AOARD project last year, I have prepared alumina templates having nano holes on the surface (Fig.1). By conventional argon ion sputtering, 50.3at.%nickel- titanium SMA was sputter-deposited on the membrane. Figure 2 is the as-deposited NiTi metallic film plus alumina membrane.

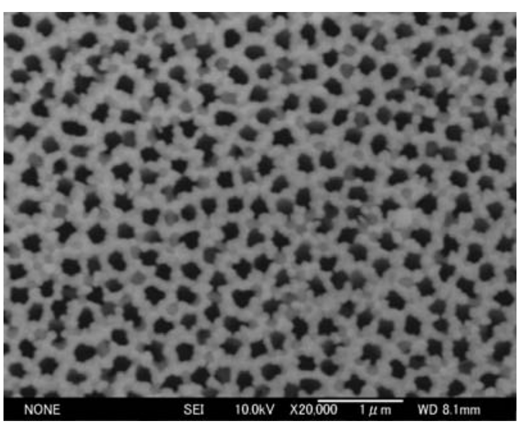

Fig.1 Porous alumina

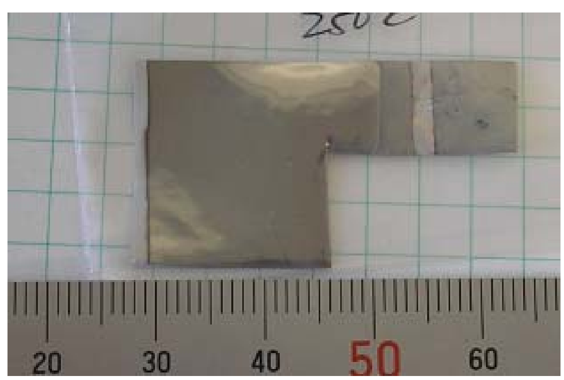

Fig.2 A sputtering deposited thin film of nickel titanium.

The alumina membrane is easy to dissolve chemically by aqueous NaOH. Then, NiTi metallic film was separated from the membrane, as shown in Figs. 3 and 4. The film was composed of columnar crystals. Figure 3 is the bottom side of the film (facing to porous alumina membrane), and Fig.4 is the cross section, where columnar crystals are seen.

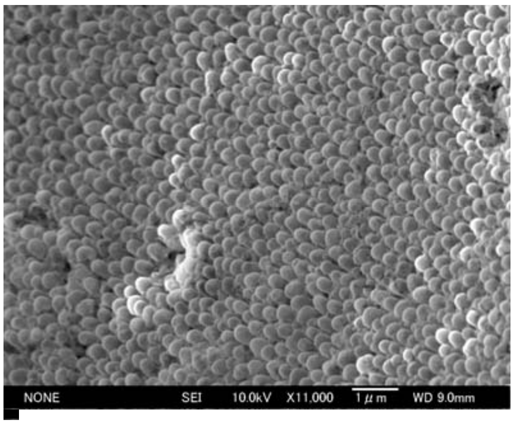

Fig3. The bottom side of sputtered film.

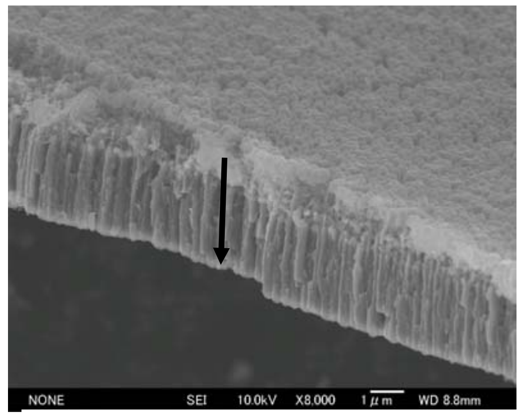

Fig4. The cross section's view. Arrow indicates the direction of crystal growth.

Figure 5 shows X-ray diffraction patterns of the sputtered film before and after annealing at 500C for 1hr. The pattern before annealing has no Bragg peak, indicating that the film was not crystallized. After the annealing was done, some sharp Bragg peaks are seen in the pattern; the peak positions are confirmed to be the same positions of those of bulk NiTi alloys.

Next, the latent heat of martensitic transformation was measured by differential scanning calorimeter (DSC). The DSC curves are shown in Fig.6. The curves show peaks indicating heat absorption and emission occurs in the annealed film. The temperatures of the peaks and their height are almost equal to the curves of bulk NiTi alloys, so that it is sure that the martensitic transformation occurs in nearly 100% volume of the annealed film.

Finally, I have found that the sputtered film is very fragile; it is easy to break it into nano-size pieces by operating conventional ultrasonic washing machine. A technical problem I am facing now is that it is difficult to get isolated fibers from the columnar crystal of Fig.4 without breaking it into particles. Figure 7 shows the SEM micrographs of disintegrated particles.

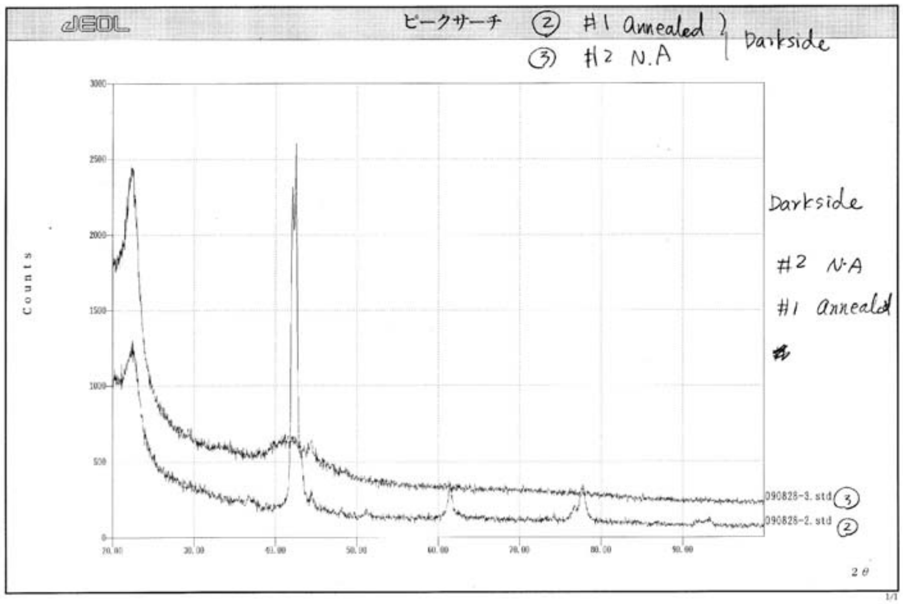

Fig.6 X-ray diffraction patterns of the as-sputtered and annealed film. As sputtered film is not crystallized, and the film after annealing at 500C shows some sharp Bragg beaks indicating that it has been crystallized.

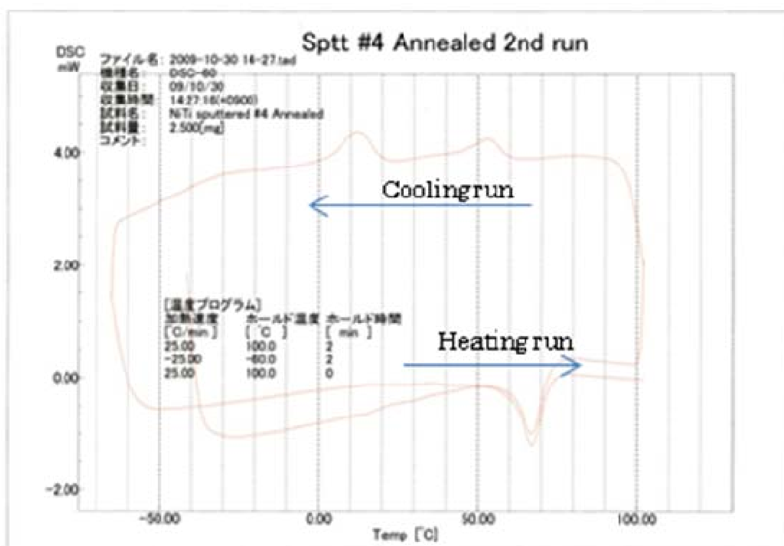

Fig.7 Differential scanning calorimeter curve of the annealed film. The curve has both end- and exothermic peaks indicating martensitic phase change occurred there. The amount of heat is equal to the bulky alloy, so it assures the material transforms almost 100% in volume.

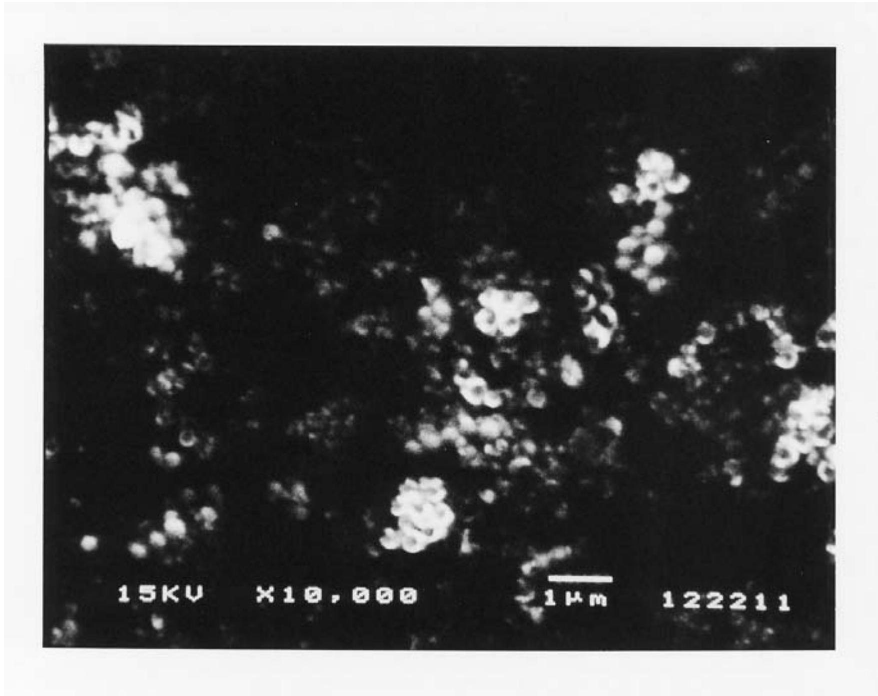

Fig.8 SEM micrograph of disintegrated NiTi sputtered film by ultrasonic wave.

- ## Conclusion

1) Sputtering growth of nano-sized columnar NiTi SMA crystal is succeeded.

2) The as-sputtered SMA film is amorphous. It can be fully crystallized and shows perfect martensitic transformation by annealing at 500C for 1h.

3) The sputtered SMA film can be disintegrated into nano SMA powders/fibers in conventional ultrasonic cleaning machine.

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